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GENERATION OF LONG TIME CREEP DATA ON REFRACTORY ALLOYS AT ELEVATED TEMPERATURES

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THOMPSON RAMO WOOLDRIDGE INC.
CLEVELAND, OHIO

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GENERATION OF LONG TIME CREEP DATA
OF REFRACTORY ALLOYS AT ELEVATED TEMPERATURES

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Prepared for
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FOREWORD

The work described herein is being performed by Thompson Ramo Wooldridge Inc. under the sponsorship of the National Aeronautics and Space Administration under contract NAS-3-2545. The purpose of this study is to obtain design creep data on refractory metal alloys for use in space power systems.

The program is administered for Thompson Ramo Wooldridge Inc. by E. A. Steigerwald, Program Manager. J. Sawyer is the Principle Investigator. H. Philleo and R. Ebert contributed to the program.

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ABSTRACT

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The first of fourteen ultra-high vacuum creep furnaces has been installed and operated. The furnace, vacuum system, and auxiliary equipment for controlling and measuring temperature, and measuring strain are described in considerable detail.

Testing has been initiated on columbium FS-85 alloy at 2000°F. After 300 hours a total extension of 0.025% was produced at an applied stress of 4000 psi.

Author

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INTRODUCTION

The object of this program is to obtain long-time design creep data on selected refractory metal alloys for use in space power systems. During this fourth quarter, the first of 14 high vacuum creep furnaces has been installed and placed in operation. The initial tests, which are being performed on columbium alloy FS-85 at 2000°F, will develop and standardize the necessary techniques for equipment operation and serve as a check between Lewis Research Lab and the Thompson Ramo Wooldridge facilities.

This report presents a detailed description of the operational procedures for the equipment and methods which are being currently employed. The items of equipment discussed in this report are:

- (1) Roughing cart
- (2) Bakeout cart
- (3) Temperature measurement and control systems
- (4) Extensometer
- (5) Water cooling system
- (6) Emergency power system.

Roughing Cart

A single roughing cart is used to service all the vacuum creep units. Its purpose is to remove most of the gas from within the vacuum chamber to a pressure at which the ion pump can be started (about 1-10 microns). The power source used for the ion pump immediately following roughing, gauges, switches, valves, and other accessory equipment required to operate the roughing system are contained in the roughing cart.

Sorption pumps provide the means of rough pumping the vacuum system to 1-10 microns without problems of vibration or back streaming of oil vapors.

The roughing cart with the adsorption pumps connected to the roughing manifold is shown in Figure 1. From right to left, the first pump is shown bare, the second has the bakeout sleeve in place, and the third has the container for liquid nitrogen in place for pumping. Above each pump is a valve connecting the pump to the manifold. Between pumps No. 2 and No. 3 is another valve for opening the system to air. The manifold extends through the cabinet at the left and has a bellows at the end to provide a flexible connection to the roughing valve attached to the vacuum system. If necessary, the manifold extending beyond the left side of the cabinet can be removed and attached to the right side.

On the instrument panel of the cart is the vacuum thermocouple gauge meter (A) connected to the thermocouple gauge (B) inserted in the manifold below the meter. This gauge is activated by the toggle switch (C). The large switch (D) activates the ion pump power supply with the high voltage leads to the ion pump extending out the rear of the cabinet. This auxiliary ion pump power supply is necessary because of the high current initially required when starting the ion pump. The toggle switch (E) is for the ion pump power supply overload. When activated, the power supply will shut off if the high voltage current becomes excessive. This switch is turned off during starting of the ion pump since high currents are required at this time. The meter (F) gives a rough indication of the chamber pressure, pump voltage, and pump current depending upon the setting of the selector switch (G). The dial gauge (H) connected to the manifold between pumps nos. 1 and 2 indicates the vacuum in inches of mercury.

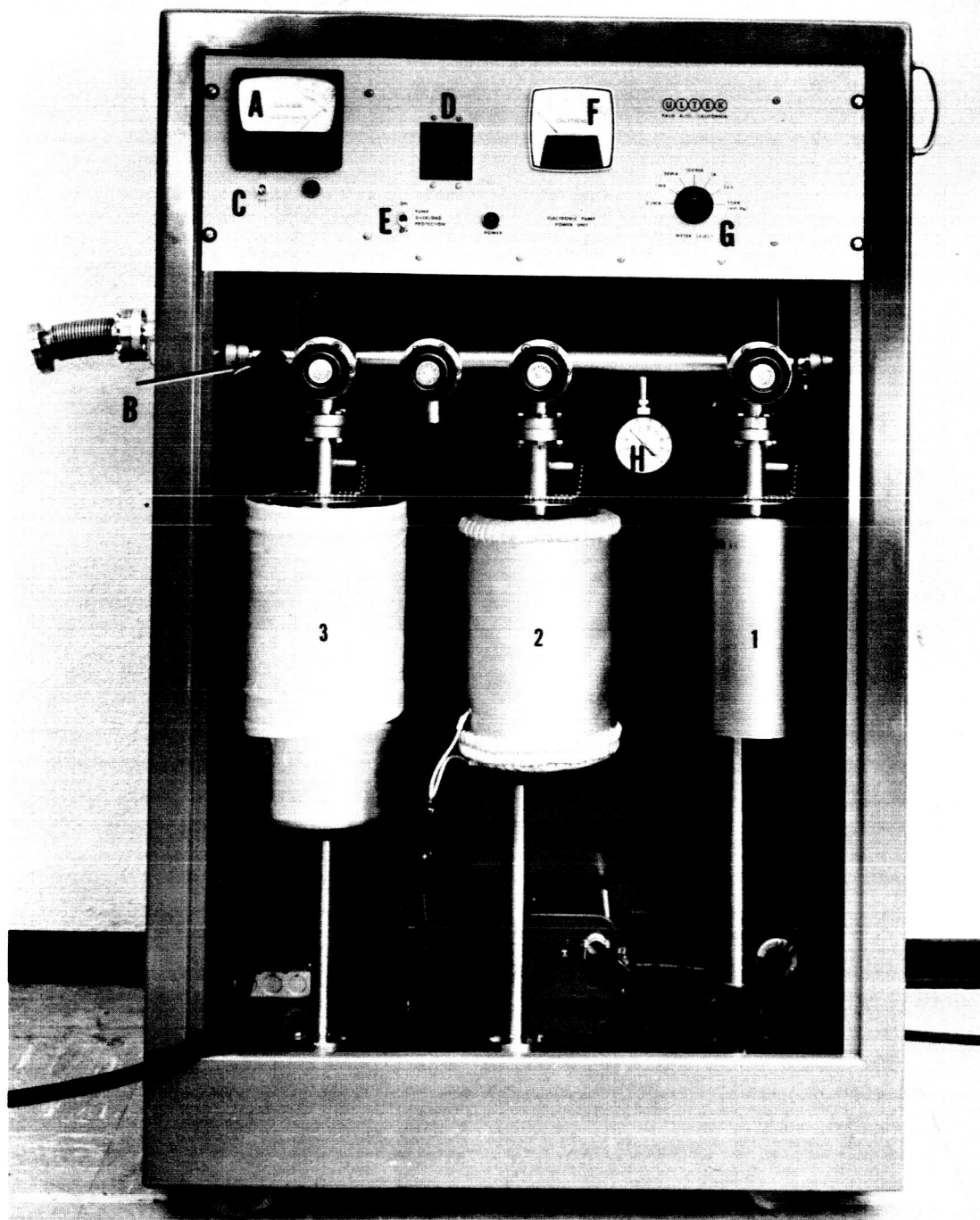


Figure 1. Roughing Cart.

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Between the body of each pump and its flange is an exhaust port closed with a rubber stopper. During bakeout of the sorption pump, the gas released causes the pressure to build up in the pump eventually blowing out the stopper. This simple device is a positive means of exhausting adsorbed gases and preventing overpressurization of a pump. The heating mantle, which fits around the pump for bakeout, raises the temperature to approximately 350°F. A four-outlet receptacle is provided in the base of the cart for the heating mantles. The cart is operated from 230 volts single phase with the necessary 120 volts being provided by an internal step-down transformer.

The procedure for using the roughing cart is as follows. The pumps are first baked out at 350°F, usually overnight. They are then cooled to room temperature by removing the heating mantle and exposing the outside of the pumps to air. If faster cooling is required, the pumps may first be cooled with water. Then the liquid nitrogen container is brought up around the pump and, with all valves closed and the stopper in place, the container is filled with liquid nitrogen. Liquid nitrogen is added periodically to bring the liquid level over the top of the pump body. After one to two hours, the liquid nitrogen becomes quiet and the pump is ready for use.

With the vacuum system and the roughing valve attached to the chamber closed, the roughing cart manifold is connected to the flange on the end of the roughing valve mounted on the front of the vacuum system (B Figure 6). This connection is made with a copper gasket. To check for leaks in this flange connection, the roughing valve is left closed and the manifold is pumped down to about 20-200 microns with pump no. 1. The valve on pump no. 1 is then closed and the pressure rise is observed on the thermocouple gauge meter (A). A fairly rapid rise even after prolonged pumping of the manifold is indicative of a leak at the roughing valve flange. A suspected leak can be verified by pumping the manifold to at least 200 microns and applying acetone to the suspected leak. If a leak is present, a sharp rise in pressure will be noted. If no leaks are indicated, the roughing valve is then opened and the vacuum chamber pumped with pump no. 1. Pumping is continued with pump no. 1 until the pressure dial gauge (H) indicates about 28 inches. Pump no. 1 is then valved off and pump no. 2 opened. Pumping is then continued until the thermocouple gauge meter indicates about 10 microns at which time pump no. 2 is valved off and pump no. 3 is opened. Once pump nos. 1 or 2 have been used and have been valved off they are not reopened to the system during the roughing cycle. When the pressure has been brought

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to 3-4 microns with pump no. 3, the overload protection switch (E) is turned off and the ion pump power supply switch (D) is turned on for a few seconds. Usually some outgassing will occur during ionization and the pressure will rise to 15-20 microns. The ion pump is then turned off and the pressure allowed to decrease to near its original value. The pressure must be below 10 microns for the ion pump to start. Extended operation of the ion pump at higher pressures would reduce life expectancy of the pump. The ion pump is again turned on for a few seconds and this procedure is repeated until the ion pump starts, as indicated by a rise in supply voltage and a decrease in ion pump current. When trying to start the ion pump, a blue glow will appear in the vacuum chamber due to ionization and when the pump starts the blue glow disappears.

When the ion pump has started and the roughing valve is closed, the roughing cart may be removed from the system by disconnecting the bellows. When the ion pump has brought the system pressure to the point where the pump current is 100 ma or less, the overload protection switch (E) is turned on. At this point the system can be allowed to pump to continually lower pressures or preparations may be made to bakeout the entire vacuum system.

Bakeout

In any ultra high vacuum system operating in the range of 1×10^{-8} mm Hg or better, it is common practice to bake out the system to drive off adsorbed gases and any volatile material from all surfaces within the chamber. If this were not done, small amounts of water, grease, or other vapors would continue to outgas (sublime or boil) for long periods of time preventing or delaying the attainment of the desired low pressure. Bakeout is accomplished by heating the vacuum system to 400°F for 8-24 hours while the ion pump is in operation.

For the vacuum system used in this program a single split-shell bake-out oven, shown in Figure 2, is used. This oven consists of two aluminum "clam" shells, each with six quartz heat lamps mounted on the inner surface. These lamps are connected in a series--parallel arrangement for operation from a 480 volt 3 phase power source. The entire oven, containing 12 lamps, provides approximately 16 KW. The shell which is of double-walled construction with insulation between the inner and outer aluminum walls, is light enough so that one person can easily move each half of the oven with the handles

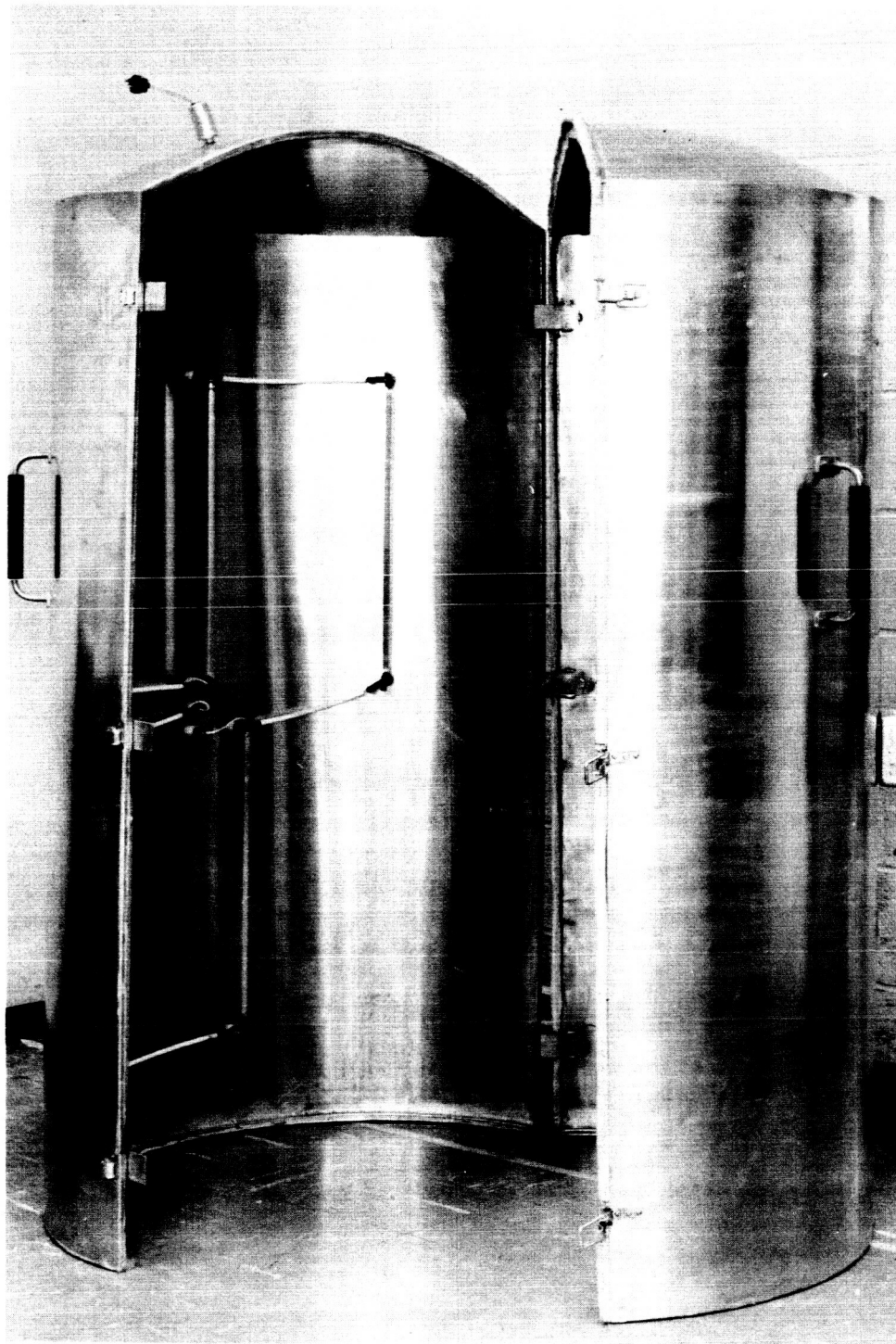


Figure 2. Bakeout Oven.

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provided. An asbestos strip is located at the junction of the two oven halves to prevent heat leaks and buckles are available to hold the two halves of the oven together when assembled around the vacuum system. A hole is also provided in the top of the oven for the temperature control thermocouple. Power and temperature control for the bakeout oven are provided by the bakeout and hoist cart shown in Figure 3.

Before baking out the vacuum system, the water is drained from the cooling jackets, the water lines are removed, and all items which might be affected by the bakeout heat are removed. This includes vacuum gauge leads and magnet, mass analyzer leads and magnet, thermocouple extension leads, shutter handle, etc. The sight port is then covered with aluminum foil to reduce heat shock of the glass. Insulating material is placed between the external weight support and weight pan, and ion pump power leads are transferred from roughing cart to bakeout cart. The oven is then placed around the vacuum system, buckled in place, and thermocouple and power leads are connected to the bakeout cart.

For this application a bakeout temperature of 400°F is used for 8-24 hours. This temperature is set on the controller, the timer is placed in position, and the pressure switch is set to prevent overload of the power supply. When the power is turned on by switch (F) the oven heats rapidly causing the system pressure to rise quickly. If the system has been opened for some time and has adsorbed appreciable water vapor, the pressure will rise sufficiently so that the power to the oven will be controlled by the pressure switch before the oven reaches 400°F. As the gas is removed and the pressure falls, the temperature will rise until control of the oven is maintained by the temperature controller. Control of the heating will then continue to be dependent on the temperature controller until the end of the time cycle at which point the timer will cut off all power to the oven.

At the conclusion of the bakeout cycle, the system is allowed to cool with the oven in place. The oven and insulation are removed only after the temperature has dropped to below 200°F. When cooled to room temperature the pressure of the system is generally in the 10^{-11} mm Hg range or lower.

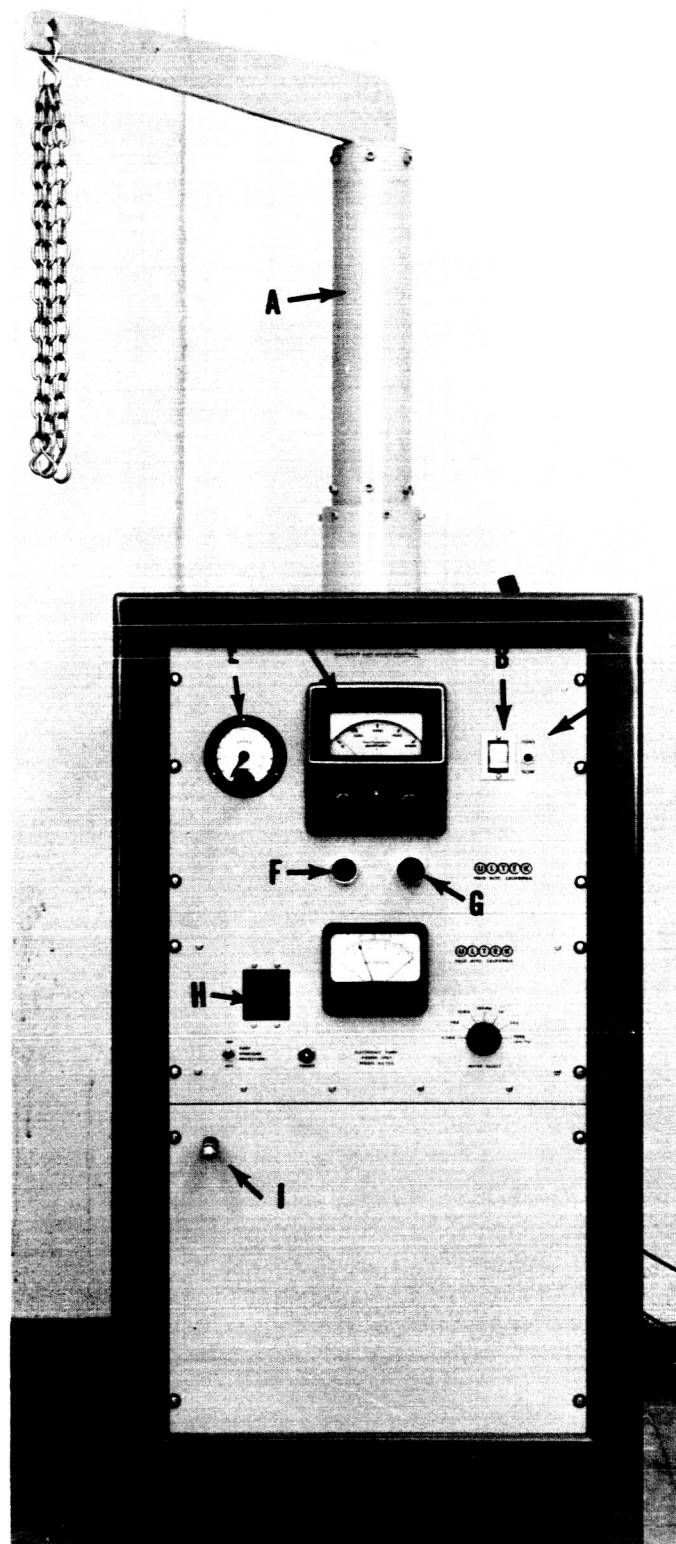


Figure 3. Hoist and Bakeout Cart.

Temperature Measurement and Control

Measurement and control of temperature under conditions of high temperature, ultra high vacuum, and very long test times require careful consideration of factors affecting stability and accuracy. Several methods are available for measuring and controlling temperature under these conditions, but no one method alone is adequate. In general, difficulties arise because the EMF output of thermocouples drifts with time at temperature and optical methods can be affected by emissivity and adsorption of radiation by vapor deposits on the sight port. Control by constant voltage input is inadequate if the conditions of temperature, vacuum, and time are such as to cause sublimation of the heating element. Control by constant power input necessitates consideration of the constancy of the ambient temperature. In view of the above considerations, a combination of methods was selected for use in this program so as to control temperatures with a minimum of variation and to measure the temperature as closely as possible to an absolute standard.

Continuous temperature control is provided by two W-3% Re/W-25% Re thermocouples connected in parallel to a Honeywell Precision three-mode controller (A), Figure 4. Parallel couples are used to provide protection against an open couple and, these couples are placed close to the heating element to minimize the response time. The controller selected has all solid state and printed circuit components for maximum life and stability. Briefly, the controller provides a thermocouple bucking voltage (B) with the EMF difference being fed to proportioning (C), reset (D), and rate (E) circuits which vary the output (F) of the controller.

In turn, the output of the controller is fed to a magnetic amplifier (not shown in Figure 4) operating from 120 volts single phase. The output of the magnetic amplifier is held to approximately 35 volts and 1 ampere to optimize the response. To achieve this, it has been necessary in some cases to provide series resistance and a dummy load in the output as shown in the schematic, Figure 5. The output of the magnetic amplifier is fed to the control winding of a 12 KVA saturable core reactor operating from 480 volts single phase. The variable output from the reactor is connected to the primary of a high current step-down transformer which, with tapped secondary, supplied the necessary power for the heating element.

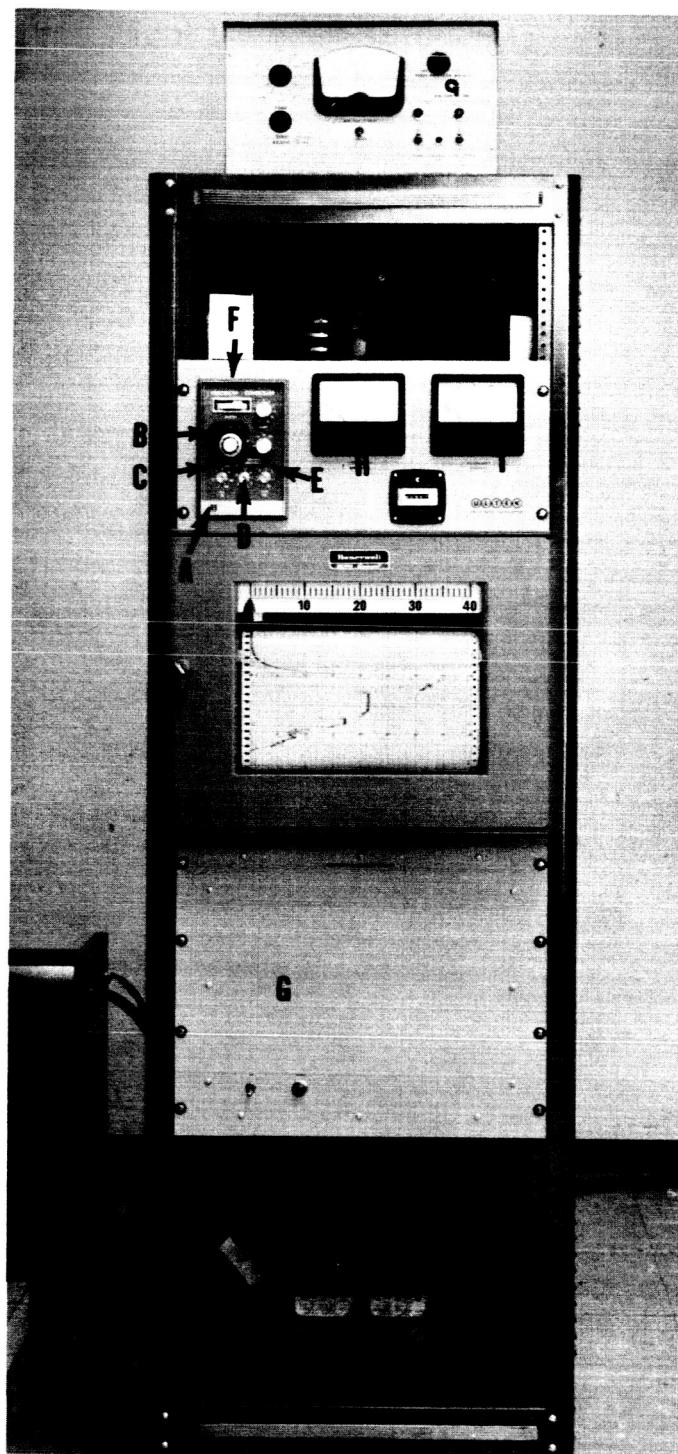


Figure 4. Temporary Console.

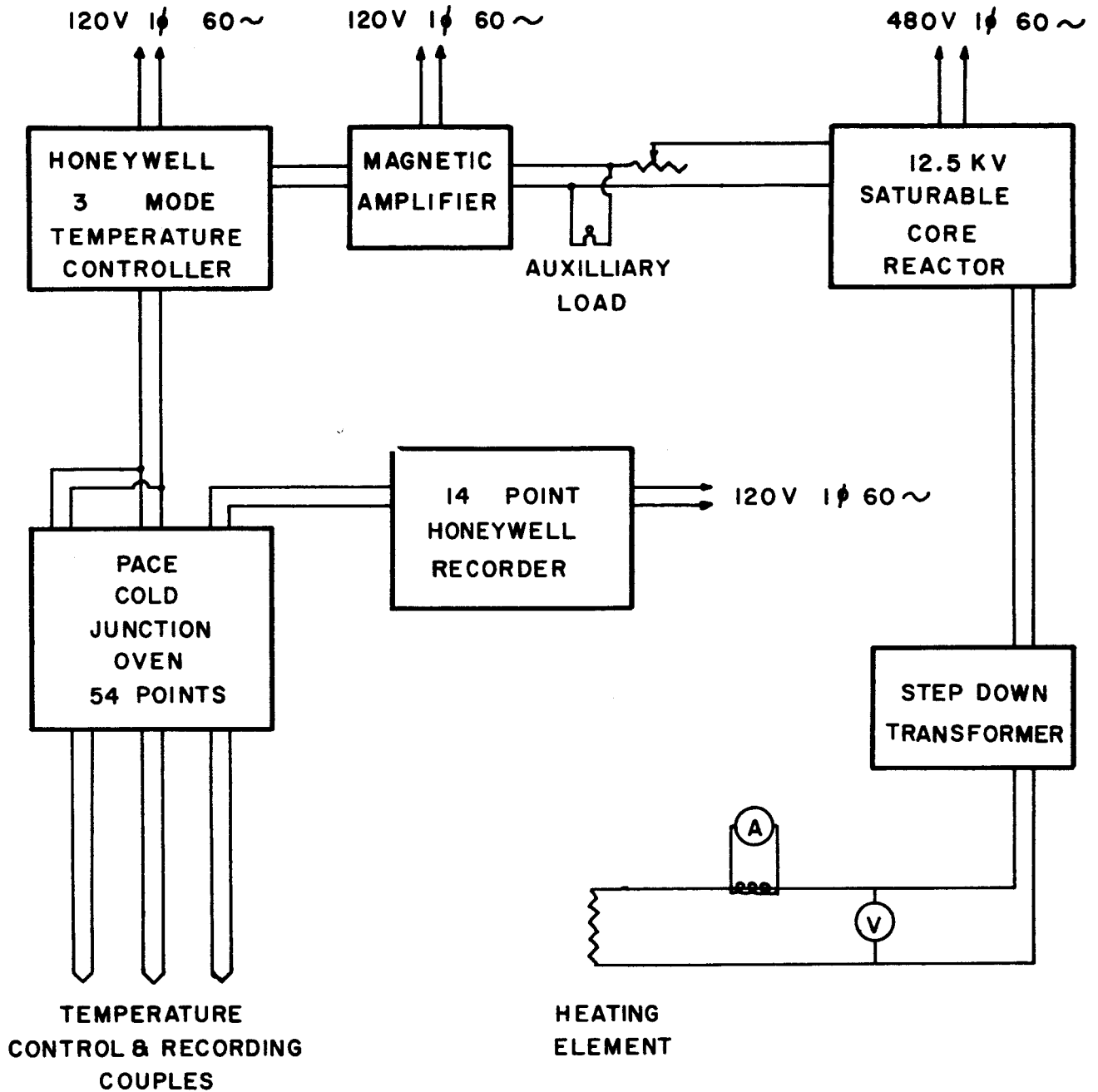


Figure 5. Temperature Control and Recording System for Vacuum Creep Units.

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To provide a reference junction of constant temperature, an oven (G) Figure 4, designed for this purpose is used since a cold junction compensator for tungsten-rhenium has not been developed. This oven operates at approximately 150°F and maintains this temperature within 1/4°F.

With this arrangement, outputs from the tungsten-rhenium thermocouples up to 40 millivolts (4000°F) can be controlled to a point where the variation of the furnace temperature does not exceed 5°F. Initial tests show that, with proper location of the thermocouples, the fluctuation over a 24 hour period is only about 1°F.

In operation, the furnace is brought to the desired temperature, as determined by a calibrated thermocouple attached to the specimen and separate measuring circuit, and then the parameters of the control system are adjusted. Setting the temperature control system consists of adjusting the series resistor and load of the magnetic amplifier, adjusting the proportioning band width, reset, and rate control, and finally adjusting the magnetic amplifier bias and controller output.

In case of failure of one thermocouple, the other will continue to control temperature since the two are in parallel. In the remote possibility of failure of both control thermocouples, the temperature will drop 30 to 50°F, depending upon the setting of the bias. Control for the balance of the test would then be maintained by adjusting the bias of the magnetic amplifier. With proper adjustment of all controls, no variation in heating element current (H) and voltage (I) can be detected. Further, any disturbance of the system caused by a sharp change in line voltage is immediately sensed and rectified with the system returning to equilibrium in less than one minute. Correction for slow changes in line voltage or ambient temperature are automatically made without any noticeable disturbance of the system. Periodic adjustments of the temperature, as determined by absolute temperature measurements, are made by adjusting the bucking voltage with the dial (B). This adjustment is fine enough that a change in furnace temperature of 1°F can be made,

To bring the specimen to a particular temperature, it is necessary to use a calibrated thermocouple connected externally to an ice bath and accurate potentiometer. For this application the calibrated thermocouple is attached to the specimen just below the gauge length test section. For a short period of time this thermocouple can be used to determine whether the specimen temperature is being maintained at the desired level, but because the EMF

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of all thermocouples drifts with time it is necessary to provide some other means of determining the temperature of the specimen. This is done by monitoring the specimen temperature with a modified IDL Pyro-650 automatic optical brightness pyrometer capable of detecting a temperature change of 1°F , Figure 6. This instrument measures temperature as a function of the radiant energy emitted by the hot specimen over a narrow wavelength band centered at 653 millimicrons. The Pyro-650 compares the energy from the specimen at the selected wavelength with the known energy from a calibrated reference lamp in the instrument. The small telescope mounted above the pyrometer (A) focuses on the same area as the pyrometer and is used as an aid in positioning the pyrometer. This is necessary because the area which the pyrometer views is less than 0.05" diameter. This pyrometer does not read temperature directly but gives a meter indication only.

In practice, as soon as the furnace has been brought to the desired temperature as shown by the calibrated thermocouple, and conditions are stabilized, the optical pyrometer is focused on the specimen at a particular spot and filters inserted such that a reading is obtained on the output meter. Tests have shown that readings can be obtained each time within $1-2^{\circ}\text{F}$ if the instrument is positioned to exactly the same spot. To achieve this, bench marks are placed on the floor and necessary measurements made so that the portion of the specimen viewed is exactly the same each time.

To avoid errors due to possible changes in transparency of the sight glass, a standard reference lamp is mounted inside the vacuum chamber. By controlling the current through the filament of this lamp it can be brought to a temperature such that the emitted energy produces approximately the same pyrometer reading as the specimen. The lamp is an argon-filled bulb so that vapor deposits do not form inside the bulb. The outside of the bulb is also shielded to prevent vapor deposits. Therefore, if the filament is heated by precisely the same current each time, changes in the radiant energy as determined by the optical pyrometer are indicative of changes in the transparency of the sight port allowing suitable corrections to be made in the observed temperature of the specimen.

In practice, the specimen temperature is measured at the start of the test with the standard WRe thermocouples attached to the specimen. The temperature of the specimen is then obtained as a meter reading on the optical pyrometer. Next, the temperature of the internal bulb is adjusted to produce approximately the same meter reading on the optical pyrometer. The lamp current necessary to achieve this condition is then accurately measured and noted for future reference. Periodically thereafter throughout the test, the temperature of the specimen is checked with the optical pyrometer. Any change noted may indicate that the temperature of the specimen has changed

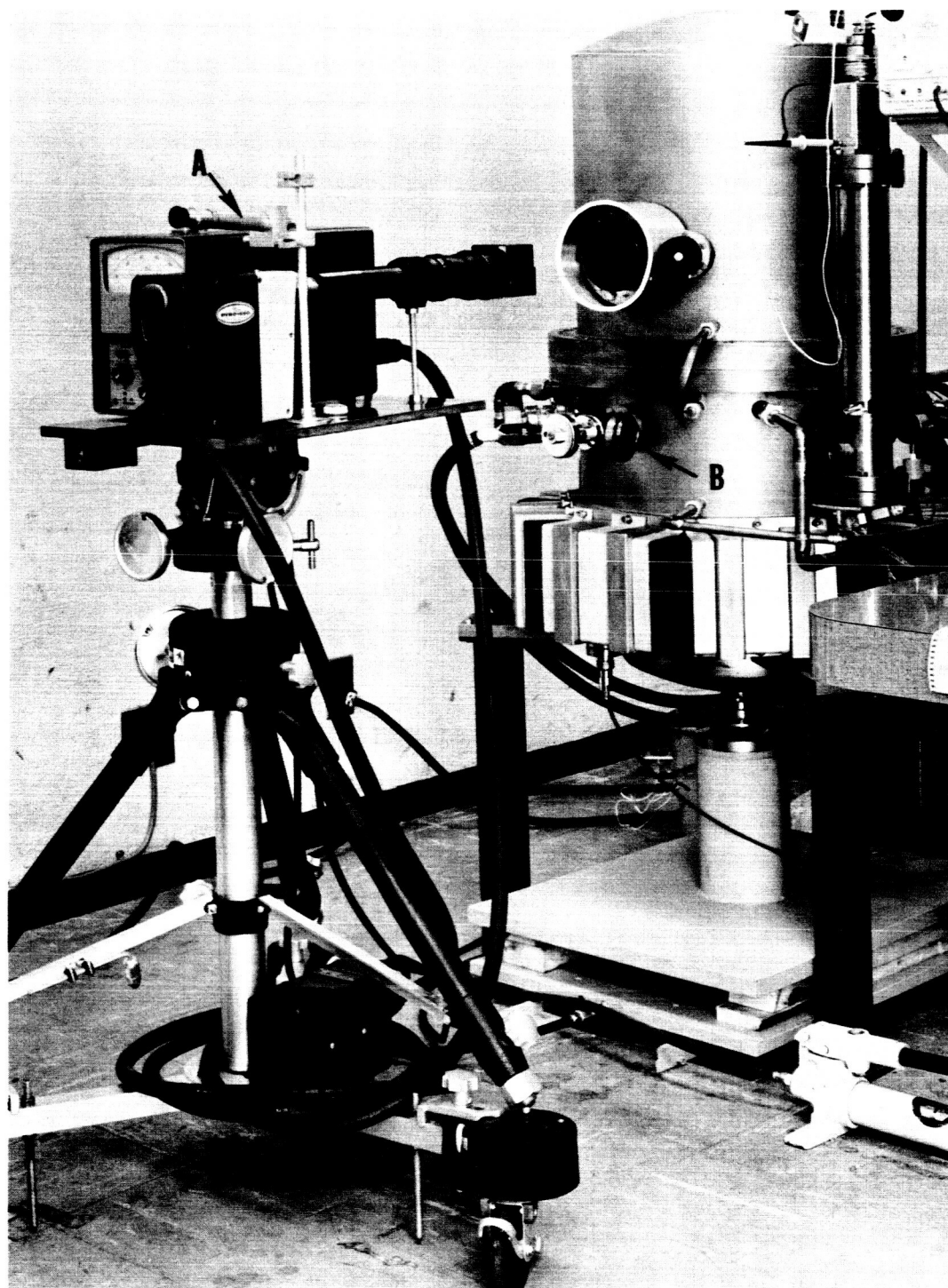


Figure 6. Optical Brightness Pyrometer.

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and/or that the transparency of the sight port has changed. The latter can be checked by passing the same current, previously noted, through the standard lamp filament and observing the meter reading. The result is that the change in specimen temperature brought about by the drift of the control couples can be detected even in the presence of vapor deposits on the sight port and suitable corrections made at the controller to bring the specimen to the desired temperature.

To assure a very high degree of reproducibility of temperature in the reference lamp, the equipment shown on the cart in Figure 7 was assembled. Its basic purpose is to provide a precisely measured current to the reference lamp. A schematic for this equipment is shown in Figure 8. Current for the reference lamp is provided by ten 6-volt storage batteries (A) connected in parallel to give 6 volts with about 700 ampere-hours capacity. A precision potentiometer (B) measures the voltage drop across a 0.1 ohm 15 amp capacity standard resistor (C). Mounted under the top shelf of the cart is another ribbon-type resistor with taps and toggle switches (D) to provide four fixed resistance; one for each of four temperature ranges: 1800°F, 2000°F, 2200°F, and 3200°F. Next to the 0.1 ohm standard resistor is a variable resistor (E) for fine adjustment of the lamp current to bring the lamp temperature to the same reading (on the potentiometer) as the specimen. The voltage drop across the 0.1 ohm resistor is then measured by the potentiometer and noted.

The potentiometer, in addition to providing a means of measuring reference lamp current is also used to measure the output of the standard thermocouple. While it is assumed that the standard couple is no longer calibrated after 50 hours, readings are periodically made to accumulate data regarding the drift of this type of thermocouple.

Extensometer

The extensometer used for measurement of strain is shown in Figure 9. This instrument consists of a double collimator system with the upper objective fixed and the lower objective movable relative to the upper by means of a micrometer screw. When the extensometer is focused on scribe marks on the specimen surface, the superimposed images of the upper and lower gauge lines are seen through the eyepiece. This is accomplished by two penta prisms which receive parallel beams of light from the upper and lower gauge marks on the specimen and combine them into a single image at the eyepiece. By

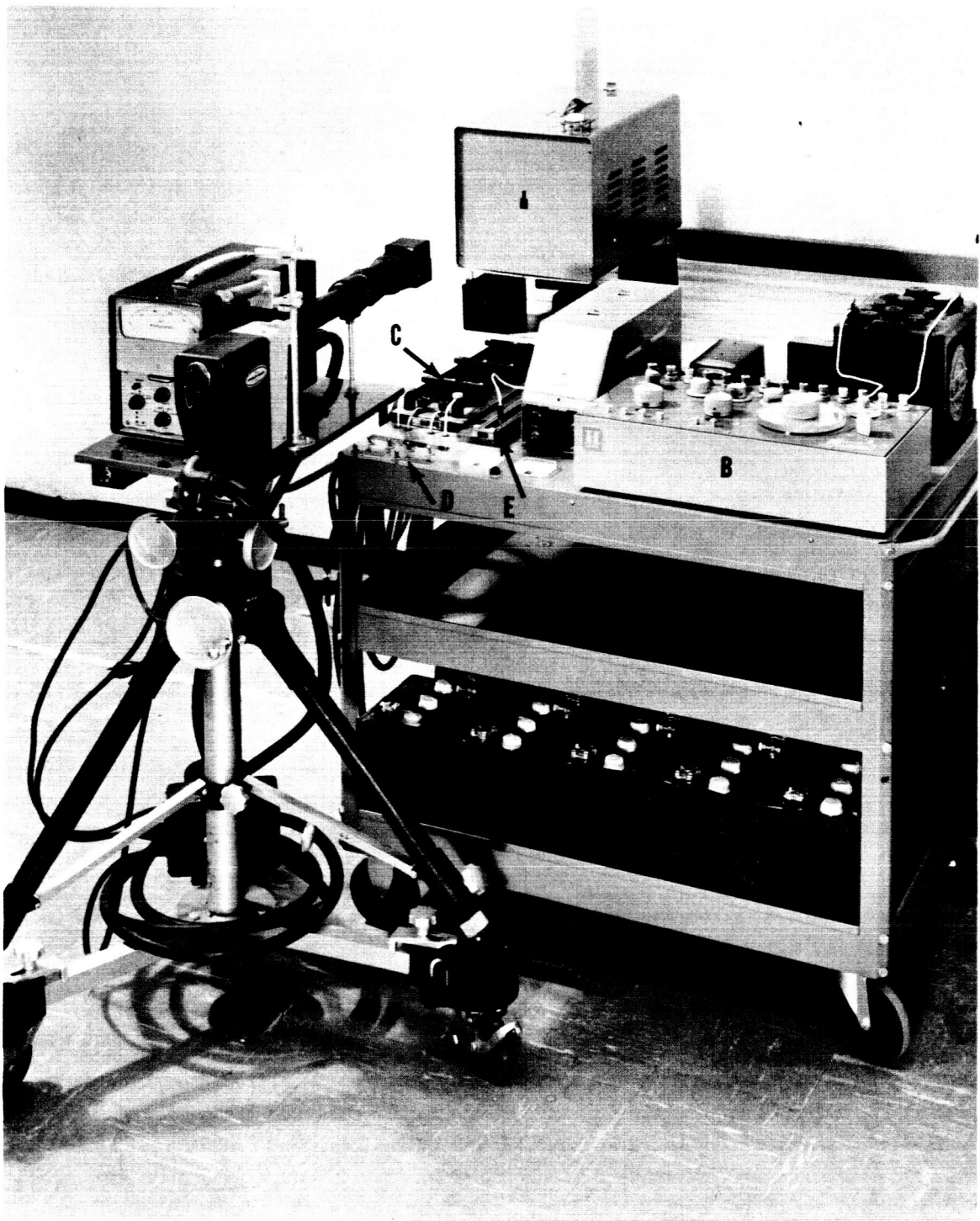


Figure 7. Calibration Cart with Optical Pyrometer.



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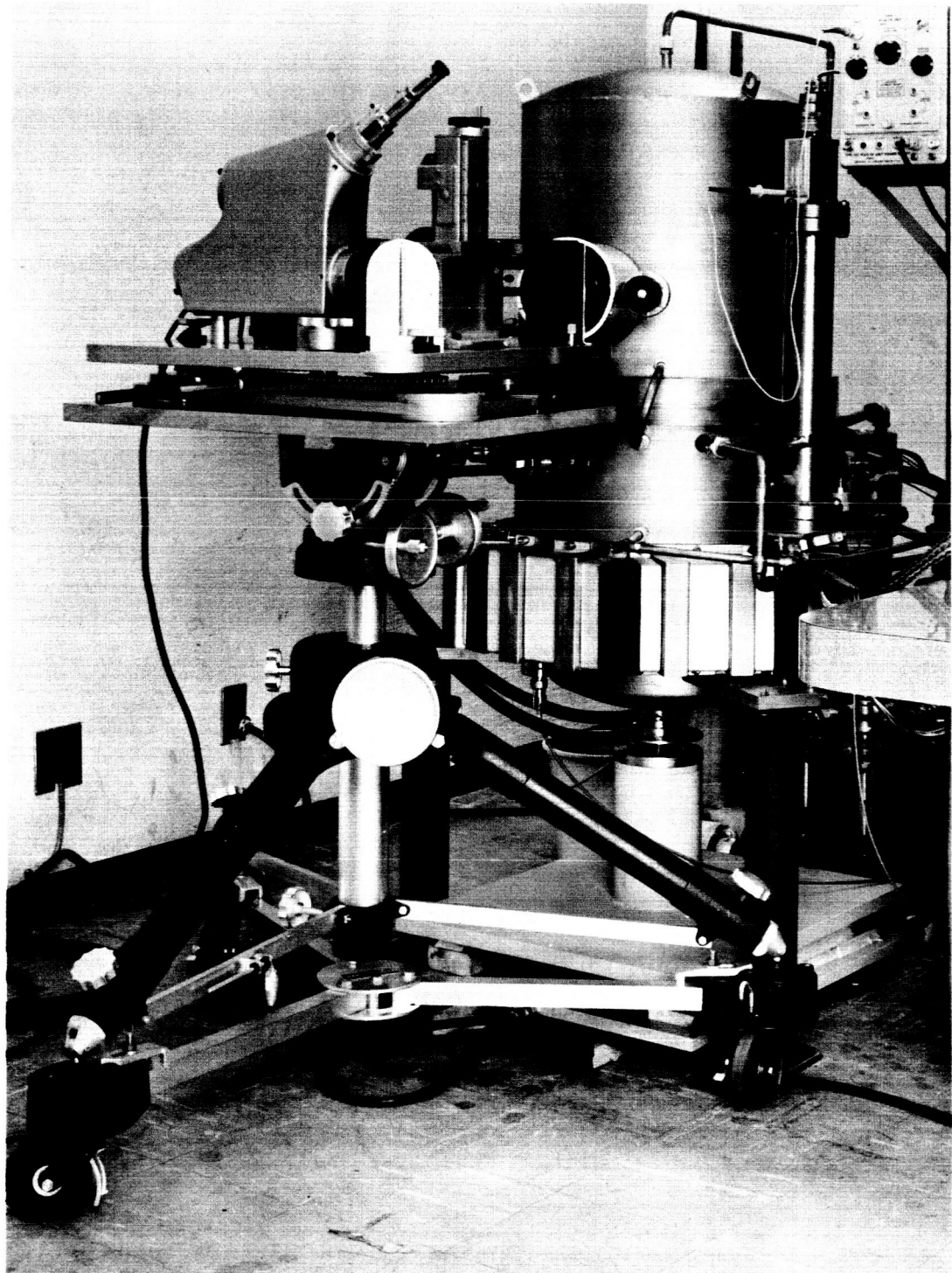


Figure 9. Extensometer in Use.

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adjusting the micrometer screw so that the image of the lower scribe mark is superimposed on the image of the upper scribe mark, the actual distance between the two scribe marks can be read directly from the micrometer scales provided on the instrument. The fine micrometer dial is calibrated to 50 microinches and has a total range of 0.75 inches.

The extensometer requires careful positioning and adjustment to obtain the best image quality and resulting high accuracy. Provisions have been made for easily duplicating position and adjustment once the correct ones have been determined, so it is not necessary to realign the entire instrument each time it is moved.

Figure 9 shows the extensometer in front of the vacuum creep system in position for measuring strain. A view of the extensometer alone is shown in Figure 10. The viewing microscope is shown at (A). The mercury arc lamp, enclosed in the lamp house (B), sends light through the condensing lens (C) to the mirror (D) from which it is reflected through the sight port to the specimen. The light reflected from the specimen returns from the top and bottom gauge mark areas, through the sight port, through the two holes in the mirror, to two prisms located in the support E. The upper beam goes to the upper stationary collimating objective and combining penta prism wedge assembly. The lower beam goes to the movable collimating objective, correcting wedge, and penta prism assembly. The micrometer (F) adjusts the position of the lower assembly to bring the two images (of the upper and lower gauge marks) together. The two superimposed images are reflected from the mirror (G) through a lens to a mirror mounted in the base of the viewing microscope to the viewer's eye.

In order to be able to quickly duplicate the position of the extensometer in relation to the specimen and sight port, two adjustable, pointed positioning screws are mounted in the Micarta base on which the extensometer rests. Two small indentations on the large flange of the creep unit receive the pointed positioning screws so that when firmly seated, duplication of location is assured.

Figure 11 shows schematically what can be seen through the viewing eyepiece during operation and adjustment of the extensometer. In theory, the distance between the gauge lines is measured by superimposing the images of the two scribe lines and then reading the micrometer. In practice it has found that the best sensitivity is obtained by bringing a particular part of one gauge line into coincidence with the other.

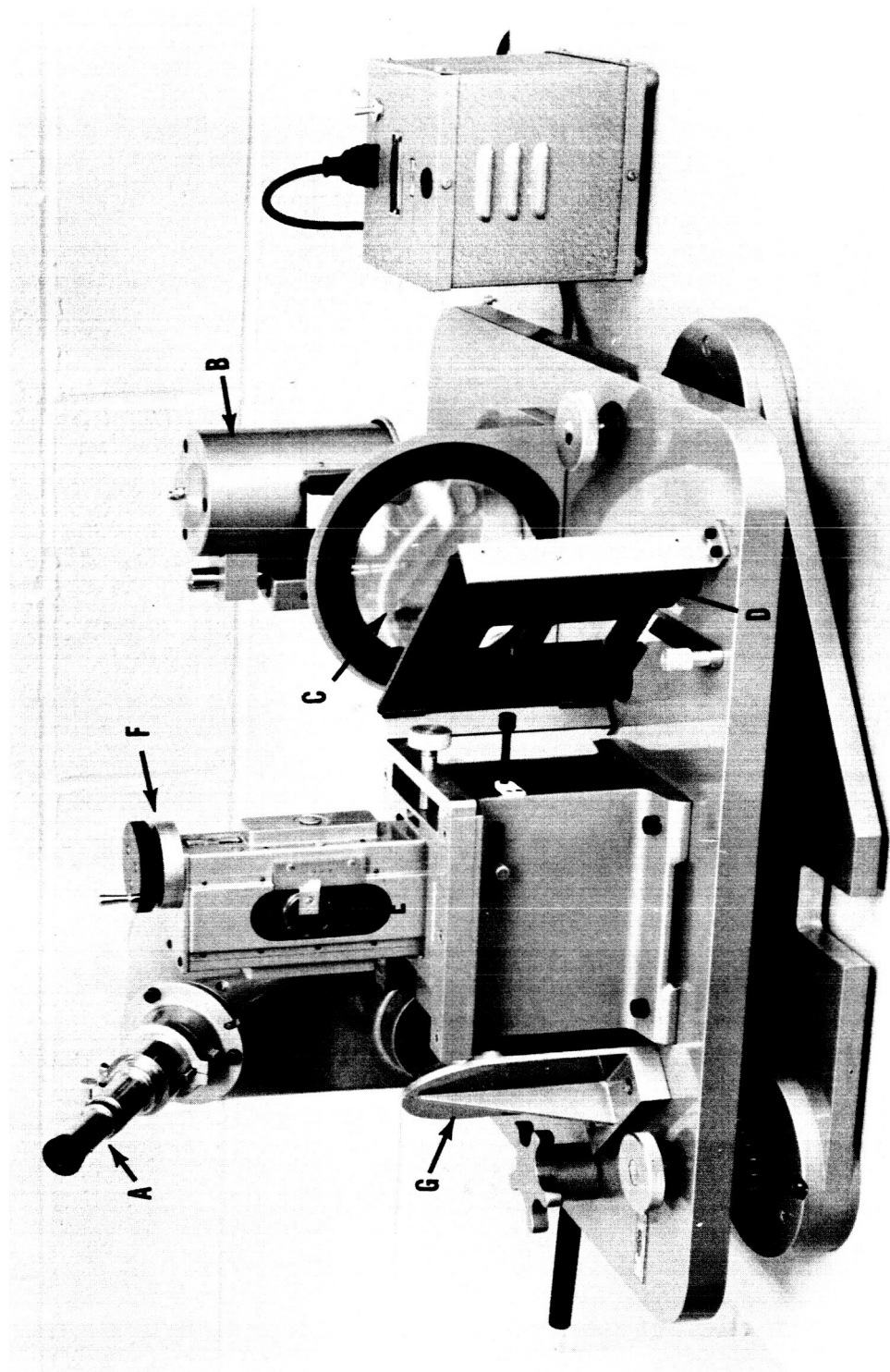


Figure 10. Optical Extensometer.

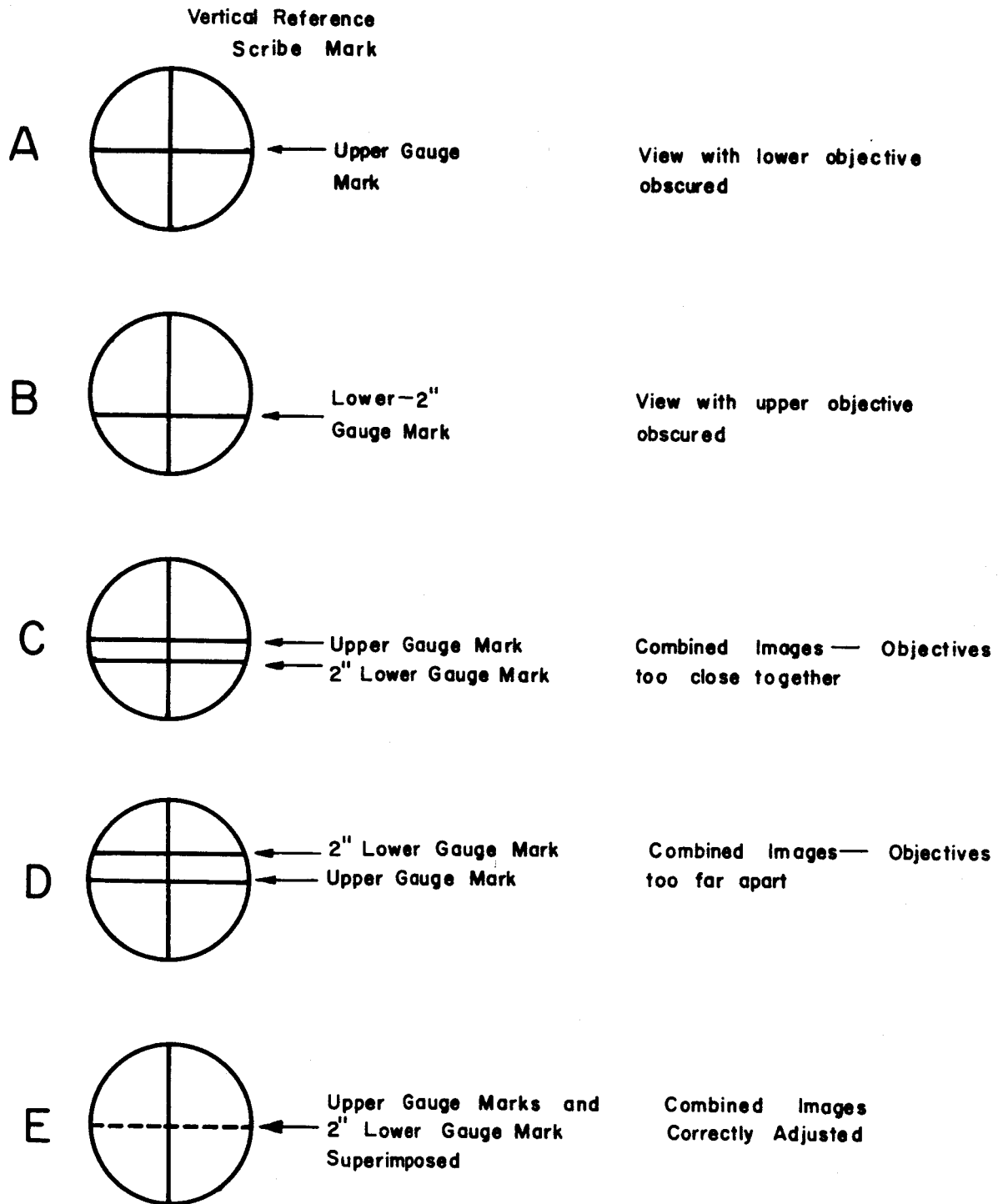


Figure 11. Gauge Marks As Viewed in Optical Extensometer.

Water Cooling System

Water cooling is used in several parts of the vacuum creep units not only to prevent overheating but to also reduce outgassing, and improve vacuum capability. The primary consideration in the design of the water cooling system is reliability since failure of this system for even a short period of time would result in damage to the vacuum creep units, loss of tests and very serious delays in the program. A secondary consideration is cost, since the volume of cooling water required for fourteen creep units is quite large.

The problem of reliability is twofold. First, there must be no interruption of water flow caused by a break in the supply main, failure of the pumps, or failure of the power to the pumps. Second, there must be no gradual decrease in water flow and heat transfer due to mineral deposits in the lines, rusting, clogging by foreign material, for the duration of the tests. These problems have been circumvented in the following ways: one, a separate recirculating water system is used which contains parallel pumps, and an emergency power system; two, the water is treated to control pH, and to prevent corrosion, deposits, and bacterial growth.

A schematic of the water cooling system for the vacuum creep units is shown in Figure 12. Pump nos. 1 and 2, Figure 13, are each of 80 gallons per minute capacity and either, is adequate for the entire system. A pressure switch located in the pipe downstream from the pumps will, if the pressure drops, switch off the acting pump, turn on the other pump, and sound an alarm. A pressure drop could be caused by a failure of the pump or drive motor, or clogging of a filter. In any case, the second pump will automatically take over the load until the trouble is corrected with the result that there should be no interruption in the flow of water. This switch-over requires less than three seconds.

When the heat load from the furnace is low, the water returns from the furnace to the sump tank directly. However, when the temperature in the sump tank exceeds a set value, the Powers valve diverts the water to the cooling tower, Figure 14, before being returned to the sump tank. The system also supplies water to the air conditioning unit serving the laboratory.

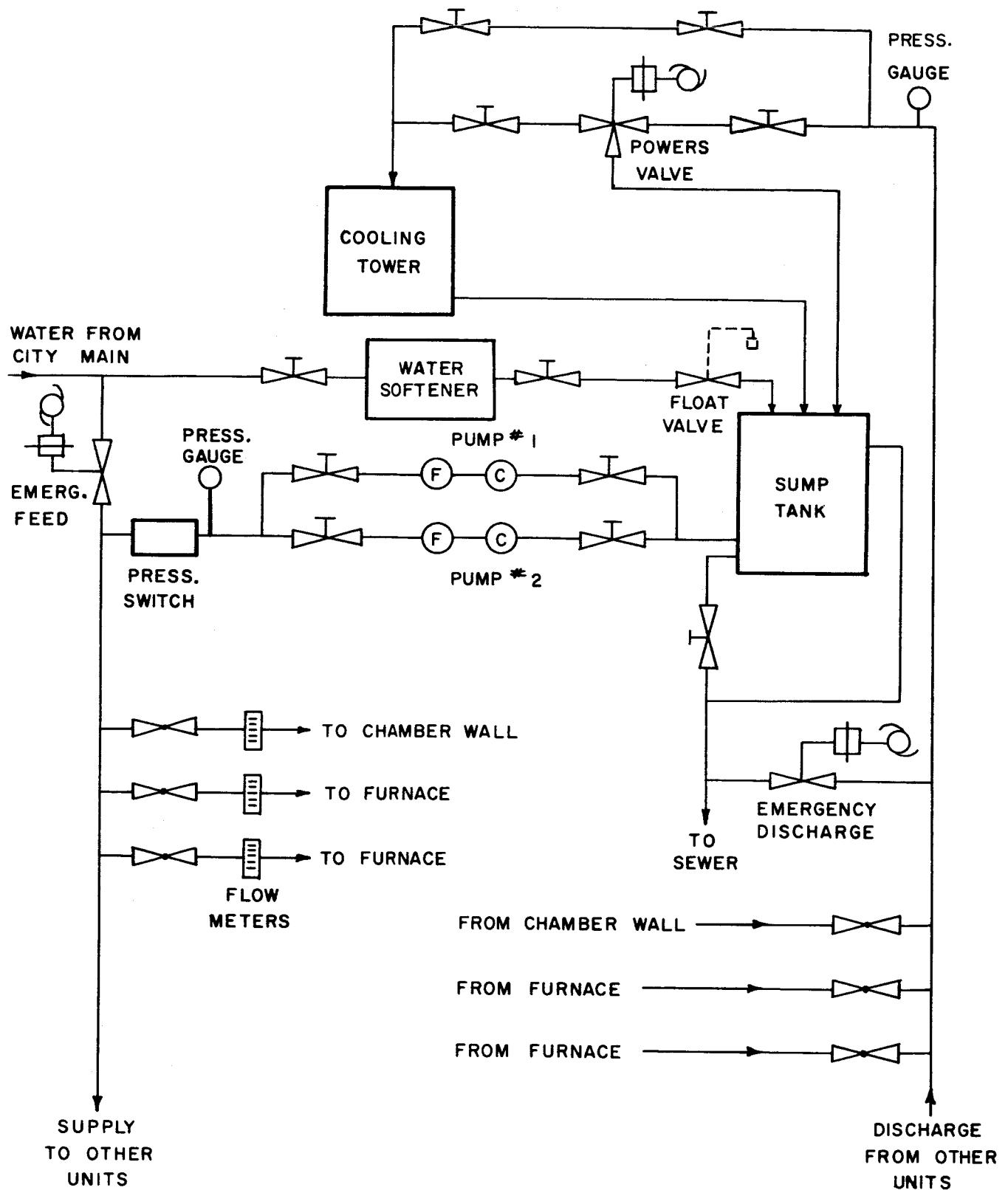


Figure 12. Water System for Vacuum Creep Units.

Pump No. 1

Pump No. 2

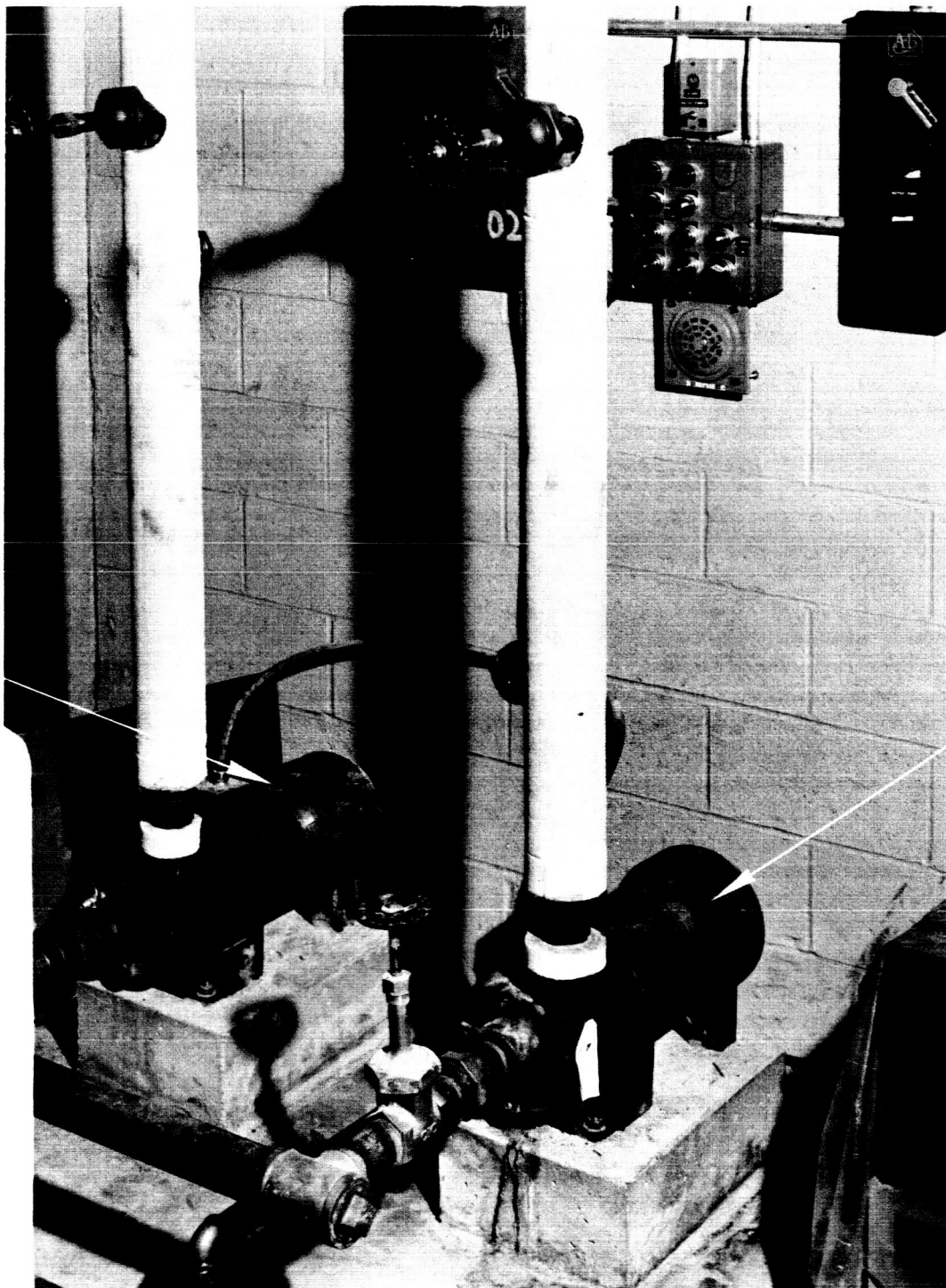


Figure 13. Water Pumps and Control Panel.

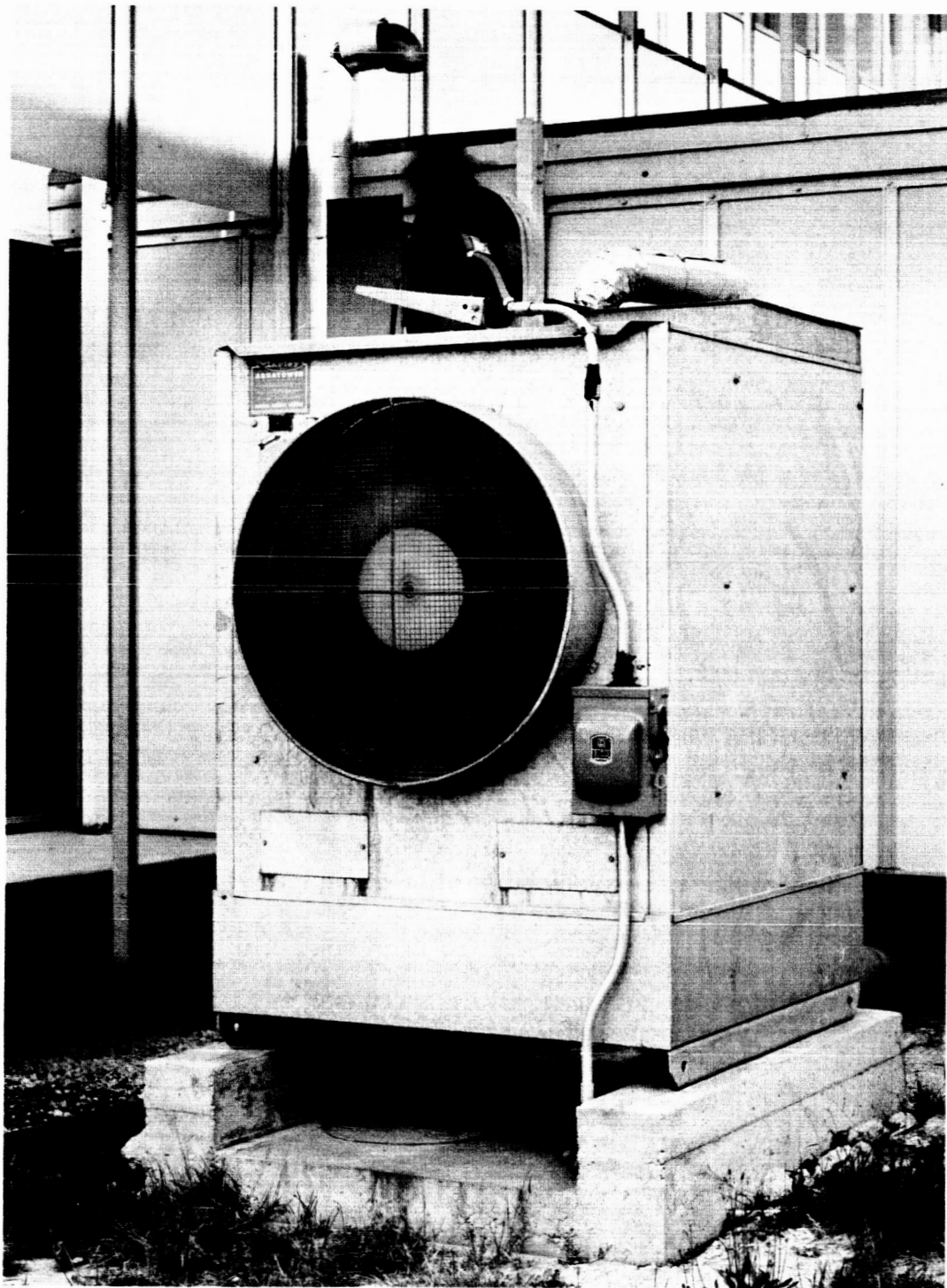


Figure 14. Cooling Tower.

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Each vacuum creep unit has two water circuits, one of which cools the vacuum chamber and the other cools the furnace coldwall. Each circuit is connected separately to the supply and return mains.

Early tests of the first vacuum creep unit at the vendor's plant showed that mineral deposits were a problem. In less than 50 hours of operation at 3500°F, mineral deposits in the furnace cooling passages had built up to the extent that the flow was restricted, the coldwall overheated, and a weld failed. Two solutions to the problem were considered, one being the use of distilled water, another being the use of softened water. The latter was selected as being the most practical in view of the amount of makeup water required to compensate for evaporation and windage losses. In either case it was necessary to treat the water to prevent corrosion since the water in passing through the cooling tower became aerated and caused excessive corrosion of the water lines. The problem was discussed with several consultants and a water treatment program was devised. The treatment used consists of periodic additions of chromate rust inhibitor, control of the pH to 7-8, and additions of biocide during the warm months to prevent algae growth in the cooling tower. These conditions are maintained by periodic tests of the water. There is still some pickup of airborne seeds, leaves, etc., at the cooling tower which are trapped at the water filters. The effect is a gradual loss of pressure, but since each pump has a separate filter, cleaning of the filter can be easily done without interrupting the water flow.

Emergency Power System

The system shown in Figure 15 was installed to provide emergency electric power because loss of power would result in heat interruption and loss of tests. This system consists of a 190 horsepower, 6 cylinder, Cummins Diesel engine driving a statically-excited Katolight, 100 KVA, 480 volt, 3 phase, 60 cycle generator. The system is designed to automatically start when the normal supply voltage falls to 80% of its nominal value with the standby power reaching full output within 5-7 seconds after starting. To protect the generator against false starts on intermittent line failure, a delay is provided so that once the standby system starts it will run for a minimum of two minutes before turning itself off despite the restoration of normal service.

The control panel, Figure 16, shows the ammeter, voltmeter, and selector switch used for monitoring the parameters of each of the three

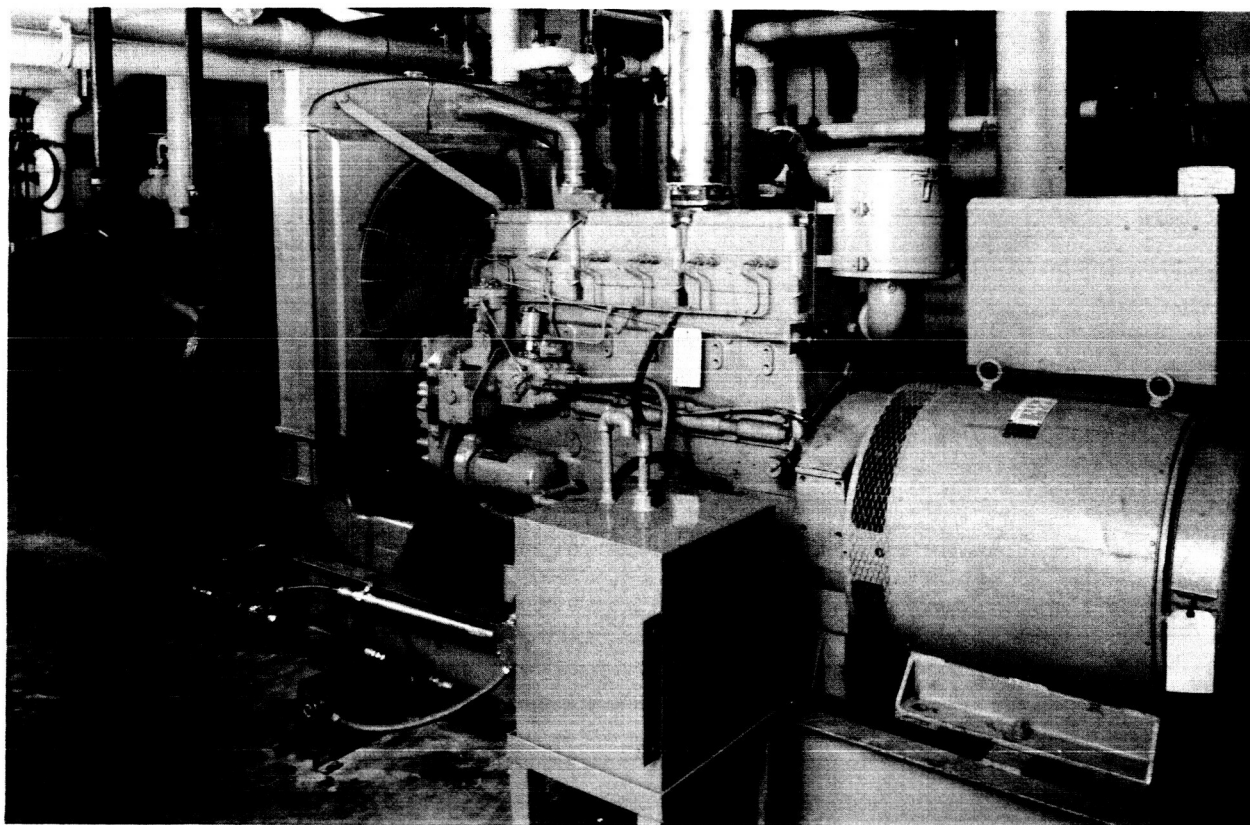


Figure 15. Emergency Power Unit.

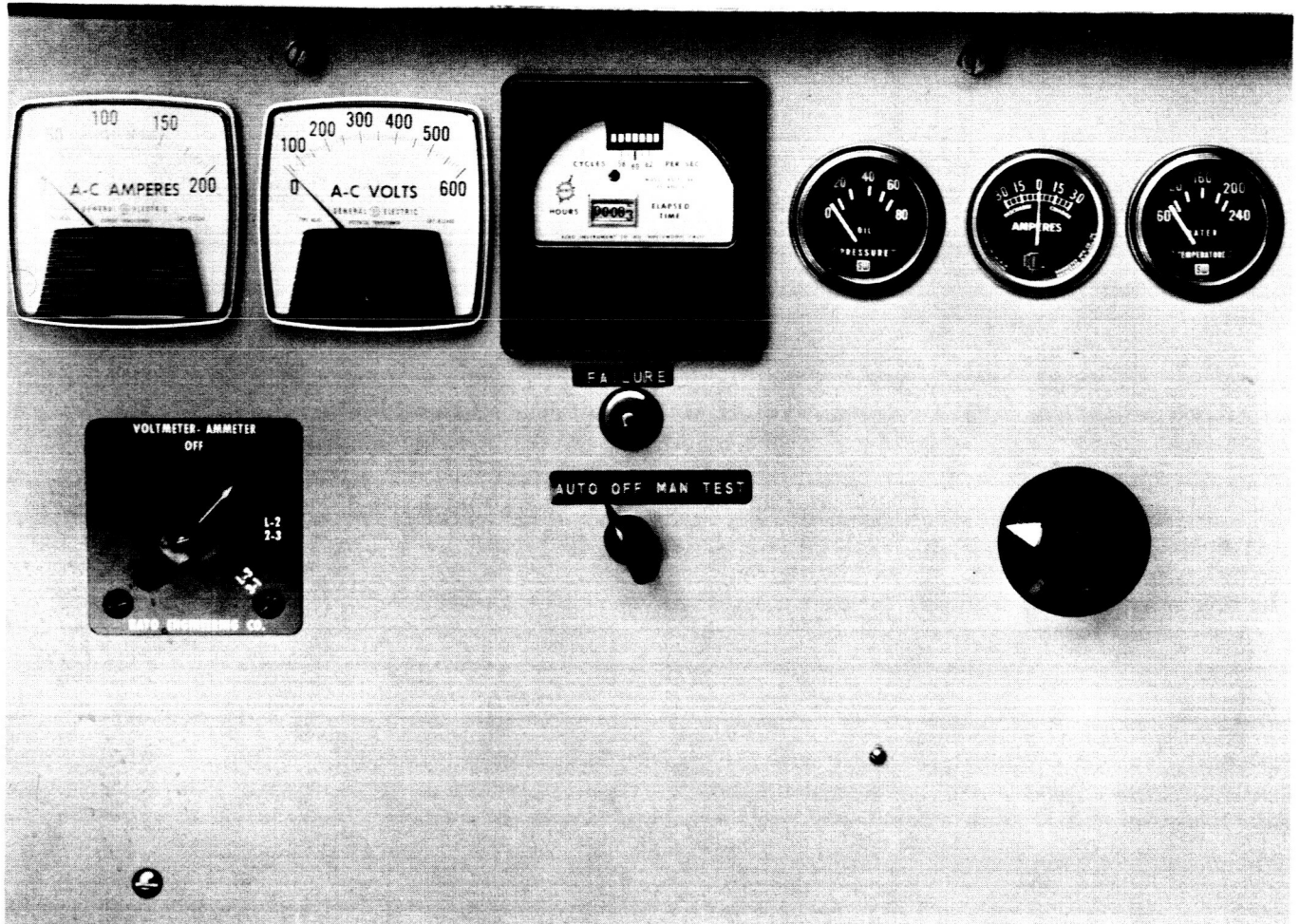


Figure 16. Control Panel for Emergency Power System.

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power phases. At the top center is a vibrating reed frequency meter for indicating the generator frequency from 57-63 cps. Once the frequency has been adjusted to a nominal value of 60 with the engine throttle control it remains quite stable. The three meters on the right indicate oil pressure, amperes charge or discharge (for the small generator which charges the starting batteries), and water temperature of the engine cooling system. The dial at the lower right is used to adjust the output voltage to match that of the nominal line voltage. The four-position switch in the center is normally left in the AUTO position for automatic operation of the power system in case of power failure. In the MAN position the engine will start, but the switching equipment to transfer the laboratory line to the diesel generator will not operate. Switching to TEST will connect the diesel generator to the laboratory line and disconnect the building supply line. When the pointer is returned to AUTO the engine stops immediately and power switching to the laboratory supply line occurs.

A second test switch, is contained in one of the cabinets and, when thrown from the AUTO to TEST position, causes the emergency power system to go through the full normal sequence of operations which would occur in case of failure of the laboratory power supply line. At full speed, the power from main building service to diesel generator service is automatically transferred. When this switch is returned to the AUTO position the engine will continue for two minutes at which time the engine will stop and the power will again transfer to the normal supply line.

Starting power for the diesel is supplied by 20 nickel-cadmium cells maintained in the fully-charged condition by a trickle charger. In addition, during operation a mechanically driven generator maintains the charge of the cells. Fuel oil for the engine is stored in a 400-gallon tank in front of the engine, Figure 15. Part of the accessory equipment not shown are various automatic air ducts and fans which are brought into operation when the engine starts to remove air heated by the diesel's radiation. Tests have shown that despite the ventilation supplied, heating of the room by the diesel's radiator is excessive in test runs where equilibrium was reached and steps are now being taken to replace the air-cooled radiator with a water-cooled heat exchanger cooled by city water.

Test Results

Much of the testing to date has been to optimize techniques, find operational difficulties in equipment, and apply corrective measures.

The first creep test was run on a sheet specimen of FS-85 columbium alloy at 2000°F and 4000 psi stress. The gauge marks on the specimen were placed at 1-1/2 and 2 inches apart in a polished area on the specimen, (see Figure 17). The calibrated thermocouple was first attached by electron beam welding the bead in a small hole drilled just below the fillet section of the specimen. This type of connection was quite brittle and was easily broken at the weld. Also, it produced a rather large heat-affected zone which could possibly lead to failure in the fillet area. Because of these considerations, another method of attaching the thermocouple was devised. Two small holes 0.020" diameter were drilled 1/16" apart just below the lower fillet section of the specimen. These holes were slightly offset so as not to weaken the section to an appreciable extent. The two thermocouple wires were brought through the holes, bent parallel to the specimen surface and the ends spot welded. A sketch of the design of attachment is shown in Figure 18. After spot welding, the weld area was swabbed with nitric acid to remove any copper deposited from the spot welded electrodes. This method of attachment produced a connection with much greater resistance to breakage and, in spite of the holes, does not produce as large an affected area as the previous method.

The first specimen was installed in the load train without removing the furnace. This was found to be time consuming because of the difficulty of feeding the specimen thermocouple through the hole in the shield pack and in a subsequent installation, the furnace was removed and the specimen installed. Temperature gradient surveys with an optical pyrometer were made on the first specimen and the results showed that the lower end of the specimen was considerably hotter than the upper end. Over a two-inch gauge length a gradient of 20-30°F at 1800°F was noted. Two sets of readings taken using a visual optical pyrometer are shown in Figure 19. One set was taken on a line (A) passing through sections of the specimen which were polished for the extensometer gauge marks and the other taken along the line (B) running parallel to (A). The vast difference in results is believed to be caused by the angle of view and the nature of the surface reflected by the specimen. In one case the specimen tended to reflect

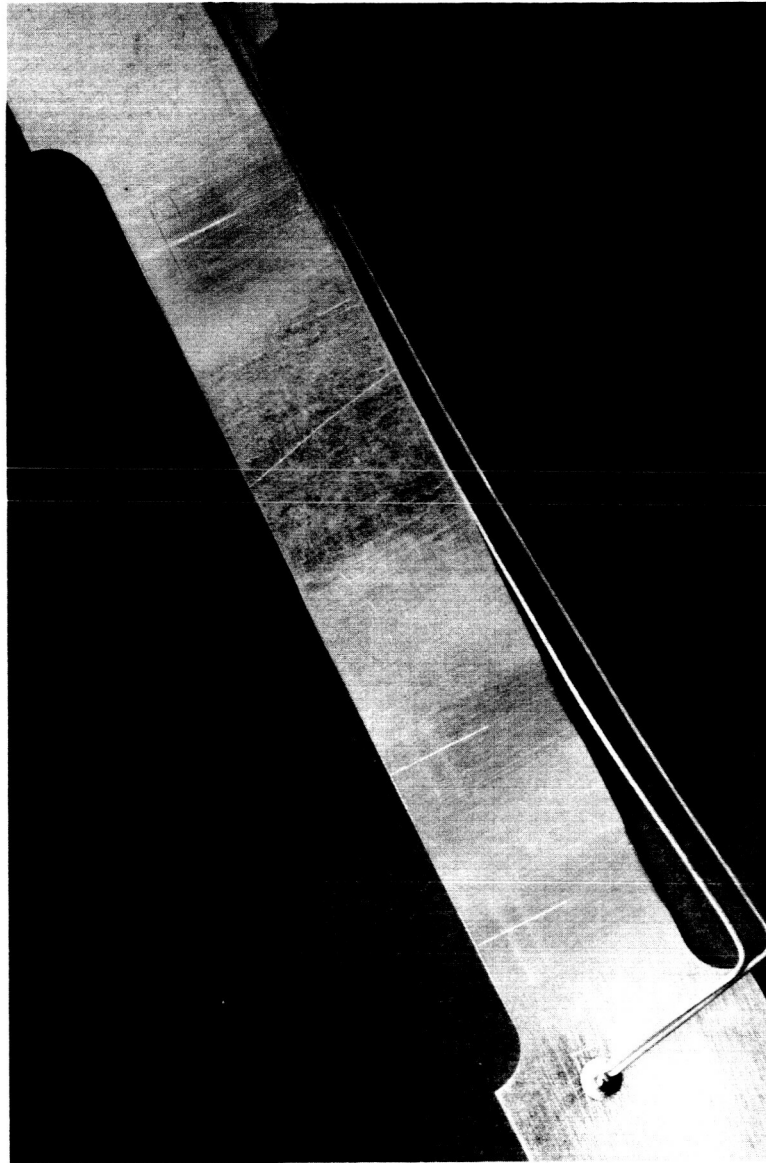


Figure 17. Initial Creep Test Specimen.



Figure 18. Creep Specimen Showing Method of Attaching Thermocouple.

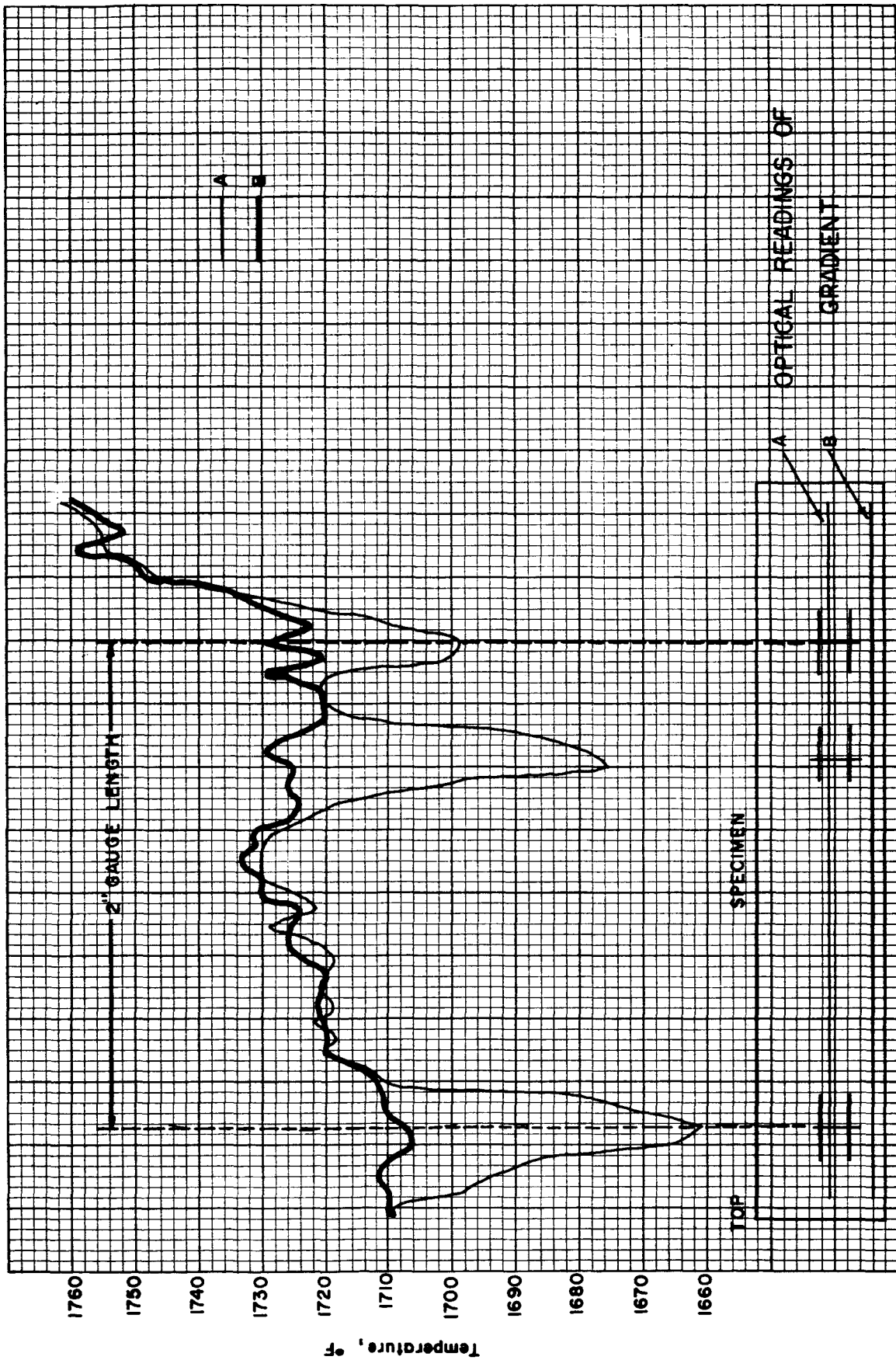


Figure 19. Temperature Distribution Along Test Specimen.

the dark opening of the furnace while in the other case the specimen was viewed at an angle so that the furnace element or heat shield was reflected into the optical pyrometer giving rise to higher and more uniform readings.

It was not possible to obtain good extensometer readings during the first test because of misalignment of the sightport with the viewing slot, shield pack slot, and heating element slot. This misalignment made it necessary to position the extensometer so far off center of the sight port that edge distortions caused the lower 2" gauge line to be blurred. Subsequently it was discovered that the specified optical flatness of the sightport was not within specifications which contributed to the problem of distortion.

An additional measurement of the thermal gradient was obtained by connecting three thermocouples at the top and bottom gauge marks and at the center of an FS-85 specimen. The couples were attached by the spot welding method previously described. To save assembly time, a "Viton" seal was used on the major flange rather than a copper gasket without significant effect on the vacuum attained. The temperatures over the specimen length were as follows: top, 1763°F; middle, 1780°F; bottom, 1790°F. These results, which are in reasonable agreement with the previous optical measurements, indicated that a serious temperature gradient existed in the furnace.

Before proceeding with further tests some corrective measure had to be taken to improve the gradient in the furnace. A partial heat shield with appropriate phase and viewing slots was spot welded to the top rim of the heating element. In addition, new end shield packs were put in the furnace. These shield packs were designed to allow the specimen to be inserted in the furnace without removing the furnace from the vacuum chamber. After adding this heat shield, the gradient over the lower half of the gauge section was 3 - 4°F at 1800°F with the middle hotter than the bottom. Note that the gradient was now reversed. Unfortunately the top thermocouple was broken during heating and the gradient between the middle and the top could not be obtained. While these results indicate that the use of an auxiliary shield is effective in changing the gradient it must be pointed out that each test involving a different specimen configuration will require that the gradient be adjusted by modification of the shield.

A test was initiated with FS-85 at 2000°F under a 4000 psi load. The specimen was inserted without removing the furnace and no difficulty was encountered. The "Viton" seal was used to allow accurate alignment of the sight ports in the system and tests showed that both the 1 - 1/2" and 2" gauge marks could be observed. Because of the "Viton" seal the system was baked at 250°F for approximately 60 hours and, when cool, pumped into

the 10^{-9} mm of Hg scale. The specimen was annealed in the creep furnace at 2600°F for 1 hour with the pressure not exceeding 1×10^{-6} mm of Hg and then cooled to room temperature.

Following the annealing operation, the specimen temperature was raised to 2000°F as determined by the calibrated couple attached to the specimen and at this temperature the pressure fell into the 10^{-9} mm of Hg range. After taking reference extension readings, the load was applied. A record of the typical data accumulated to approximately 300 hours is presented in Table I and Figure 20.

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TABLE ICREEP TEST DATAColumbium Alloy FS 85, 2000°F, 4000 psi.Optical Readings
(Microamperes)

<u>Hrs.</u>	<u>Specimen</u>	<u>Internal Std.</u>	<u>Pressure mm Hg</u>	<u>Length Inches</u>	<u>Remarks</u>
-	-	-	6.1×10^{-11}	2.00163 ⁽¹⁾ 1.50525	cold - no load
-	-	-	-	2.01925 1.51838	2000°F - no load
1	-	-	1.4×10^{-9}	2.01994 1.51860	2000°F - loaded avg of 0.13 and 0.6 hrs.
23	47-50	55	1.4×10^{-9}	-	
96	48-50	-	6.3×10^{-10}	2.02004 1.51883	
161	62-66	52	5.4×10^{-10}	2.02012 1.51888	temp. decreased 3°F
190	52	-	5.4×10^{-10}	2.02016 1.51890	
304	49	-	5.2×10^{-10}	2.02051 1.51934	

(1) Average ten readings

Accuracy and reproducibility of readings with the optical extensometer are improving as procedures and techniques are developed and refined. Some of the early readings, however, show more scatter than is desirable, especially in the 1-1/2 inch data. Thus far it appears that a permanent extension has occurred which, when extrapolated to 1000 hours, may amount to

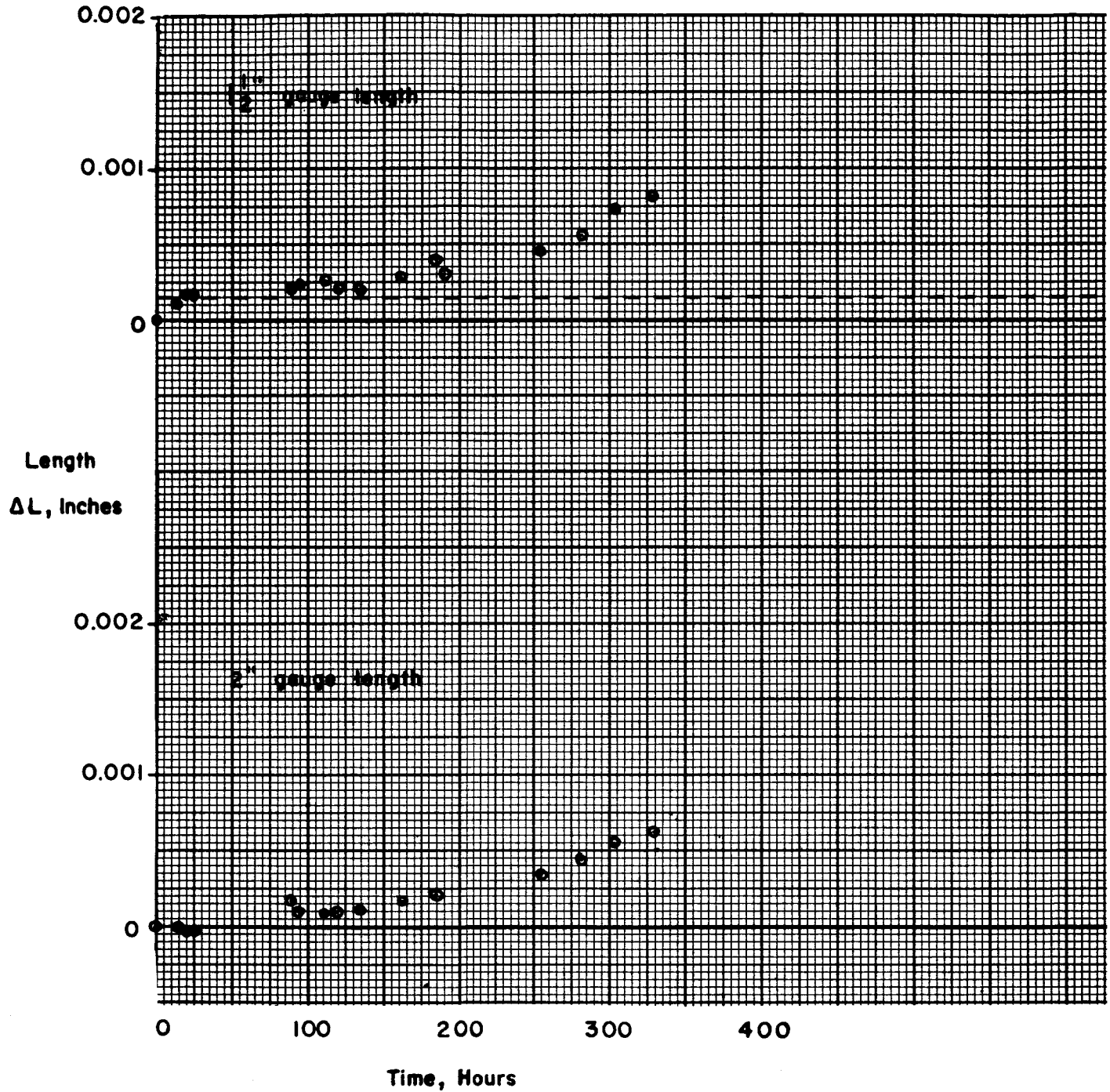


Figure 20. Creep Curves for Columbian Alloy FS-85 Specimen #2, 2000° F, 4000 psi, Vacuum Approximately 5×10^{-10} mm Hg.

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approximately 0.3% creep. Further measurements will establish this point more conclusively.

Measurements of temperature during the 300 hours test time showed that after approximately 150 hours, slight clouding of the sight port occurred amounting to a 1°F change in apparent temperature. In addition, the optical readings on the specimen indicated a 3°F drift of the thermocouples necessitating a change in the controller to restore the temperature to 2000°F. A test of the change in the furnace temperature showed that the furnace cycled approximately 2°F over a period of 45 minutes in a regular manner. This fluctuation was noted to correspond with a small change in the cold junction oven temperature. This fluctuation is being examined to determine the cause and make suitable correction to reduce the temperature fluctuation to 1°F or less.

Status of Test Alloys and Equipment

The following is a status summary of the alloys and equipment which are to be used for this program:

<u>Item</u>	<u>Form</u>	<u>Source</u>	<u>Shipping Date</u>
FS-85	sheet	NASA	received and currently being tested
TZM	disc forging	Climax Molybdenum	received
TZC	plate	General Electric	9/4/64
ST-222	plate	Westinghouse	under negotiation
Cb-132-M	plate	Universal Cyclops	9/18/64
AS-30	plate	General Electric	9/19/64
W	sheet	Universal Cyclops	7/12/64
W-25%Re	sheet	Wah Chang	7/10/64
Sylvania-A	sheet	Sylvania	11/2/64
console		Ultek	8/2/64
5-creep units		Ultek	8/3/64
8-creep units		Ultek	9/18/64

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Work in Progress

The test of FS-85 sheet at 2000°F and 4000 psi will be continued for a maximum test time of 1,000 hours. During this time efforts will be directed towards improving techniques of temperature and extension measurement.

A water cooled heat exchanger has been ordered for the diesel engine used to power the emergency electric power generator. The radiator now on the engine heats the laboratory building too much.

Electric power lines for the console and five more vacuum creep furnaces are being installed in the creep laboratory. Water and drain lines for the furnaces are also being installed.

Further tests are underway to measure and adjust the temperature gradient along the specimen.

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